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IS 8488 (1977): Phosalone, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]

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Indian Standard

SPECIFICATION FOR PHOSALONE, TECHNICAL

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

rice Rs **6·00**

September 1977

Indian Standard

SPECIFICATION FOR PHOSALONE, TECHNICAL

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(*Continued on page* 10)

Indian Standard

SPECIFICATION FOR PHOSALONE, TECHNICAL

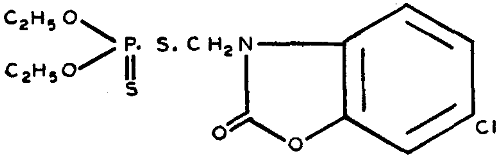
0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 June 1977, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Phosalone is a non-systemic insecticide and acaricide and is used for controlling a broad range of pests of agricultural crops.

0.3 Phosalone is the accepted common name by the International Organization for Standardization (ISO) for the pesticidal chemical S-(6-chloro-2-oxobenzoxazolin-3-yl) methyl 0, 0-diethyl phosphorodithioate. The empirical and structural formulae and molecular mass of phosalone are as given below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_{12}H_{15}Cl\,NO_4PS_2$		368



0.4 In the preparation of this standard due consideration has been given to the provisions of the Insecticides Act, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

---

*Rules for **rounding off numerical values (** ***revised*** **).**

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for phosalone, technical.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of white to pink or yellow crystals or powder with typical odour of garlic.

2.2 **Solubility** — 10 percent (*m/v*) solution of material in acetone shall be clear and 2 percent (m/v) solution of material in acetone shall not be darker than the colour of 0·000 2 N iodine solution.

2.3 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR PHOSALONE, TECHNICAL

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO	
			Appendix of This Standard	Cl No. of IS : 6940-1973*
(1)	(2)	(3)	(4)	(5)
i)	Phosalone content, percent by mass, *Min*	93	*A*	—
ii)	Melting point	42” to 48°C	—	6
iii)	Moisture content, percent by mass, *Max*	1	B	—
iv)	Acidity (as H_2SO_4), percent by mass, *Max*	0·1	—	11.3.2
	OR			
	Alkalinity (as NaOH), percent by mass, Max	0’1	—	11.3.3

*Methods of tests for pesticides and their formulations.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in clean and dry containers made of mild steel or tinplate or fibre board or double hessian jute bags (*see* IS : 8115-1976*) or DW tarpaulin laminated jute bags (*see* IS: 8117-1976†) or HDPE woven sacks (see IS: 8069-1976‡). The containers shall also comply with the general requirements as stipulated in 2 of IS : 8190 (Part I)-1976§.

---

*Specification for double hessian jute bags for pesticides.

†Specification for DW tarpaulin laminated jute bags for pesticides.

‡Specification for high density polyethylene woven sacks for packing pesticides.

§Requirements for packing of pesticides: Part I Solid pesticides.

9.2 Marking — The containers shall bear legibly and indelibly the following information in addition to the information as is necessary under the Insecticides Act and Rules:

a) Name of the material,

b) Name of the manufacturer,

c) Date of manufacture,

d) Batch number,

e) Net mass of contents,

f) Phosalone content percent (*m/m*), and

g) The cautionary notice worded as in the Insecticides Act and Rules.

3.2.1 When packed in bags, the pictorial marking for ' USE NO HOOKS ' and ' DO NOT PUNCTURE ' as specified in IS: 1260 (Part II)-1973* shall be stencilled on the bags.

3.2.2 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by IS1 for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the IS1 Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn according to the method prescribed in ' Indian Standard Methods for sampling of pesticides and their formulations ' (*under preparation*).

NOTE -*Till* such time the standard under preparation is published; the matter shall be as agreed to between the parties concerned.

5. TESTS

5.1 Tests shall be carried out by the methods referred to in col 4 and 5 of Table 1.

5.2 **Quality of Reagents** — Unless specified otherwise, pure chemicals and distilled water (see IS :1070-1977†) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

---

*Pictorial markings for handling and labelling of goods: Part II General goods.

†Specification for water for general laboratory use (*second revision*).

APPENDIX A

[*Table 1, Item* (i)]

DETERMINATION OF PHOSALONE CONTENT

A-O. GENERAL

A-0.1 Either of the two methods, namely, gas chromatographic method and spectrophotometric method may be used for determination of phosalone content.

A-I. GAS CHROMATOGRAPHIC METHOD

A-l.1 Principle of the Method — The method consists of injecting a sample with an internal standard in a known proportion into a gas chromatograph and determining the area under each peak. The area under the peak is proportional to the mass of the sample. By comparison of this area with that of the standard, the percentage purity of the sample is determined.

A-I.2 Apparatus

A-1.2.1 *Gas-Liquid Chromatograph* — equipped with a recorder and a disc integrater and fulfilling the following conditions:

Column length, mm	400
Detecter	Thermal conductivity type with 170 mA current
Column temperature	240°C
Injection port temperature	270°C
Detecter temperature	275°C
Carrier gas	Helium or hydrogen
Flow rate, l/h	3·6
Chart speed, mm/h	760

A-1.2.2 *Column* — consisting of 400 mm, stainless steel (designation 04Cr14Ni10) tubing, of 6·35 mm outer diameter, packed with 10 percent GE-SE-30 on 125 to 250 micro Embacel DMCS treated.

A-1.2.3 *Micro-syringe* — 10 µl capacity.

A-I.3 Reagents

A-1.3.1 *Diethyl Ether*

A-1.3.2 Internal Standard Solution — 2,4, 5-T butylglycolic ester, free from any impurity likely to interfere with phosalone under the chromatographic conditions.

NOTE — Any other standard may be used if this is clearly differentiated from phosalone and the other constituents that may appear on the chromatogram.

A-1.3.3 *Standard Reference Phosalone*

A-1.4 Procedure

A-1.4.1 *Preparation of Test Solution* — Weigh accurately 0·15 to 0·17 g of the sample and 0·07 to 0·08 g of the internal standard into separate weighing bottles. Transfer quantitatively to 10 ml volumetric flask with diethylether and adjust to mark with diethylether.

A-1.4.2 *Preparation of Standard Solution-Weigh* accurately 0·15 to 0·17 g of the standard reference phosalone and 0·07 to 0·08 g of the internal standard into separate weighing bottles. Transfer quantitatively to 1 O-ml volumetric flask with diethylether and adjust to mark with diethylether.

A-1.4.3 *Analysis of Sample* — Check the chromatograph for thermal and flow equilibrium. Inject 2 µl of the standard solution (A-1.4.2) and calculate '*R*' for each chromatogram

where

$$R = \frac{\text{phosalone peak area}}{\text{internal standard peak area}}$$

NOTE — If *R* has not a constant value, repeat injections till '*R* is constant within limits of experimental error.

A-1.4.3.1 Inject alternatively 2 µl of the test solution (A-1.4.1) and the standard solution (A-1.4.2) at least thrice.

A-1.5 Calculation

A-1.5.1 Phosalone content, percent by mass $= \frac{F_{spl}\,T}{F_{std}}$

where

$$F_{spl} = R_{spl} \times \frac{\text{mass of the internal standard in test sample solution}}{\text{mass of the material taken for preparation of test solution (A-3.2)}}.$$

$$F_{std} = R_{std} \times \frac{\text{mass of the internal standard in standard solution}}{\text{mass of the reference standard phosalone taken for the preparation of the standard solution}},$$

R_{spl} = the mean value of the '*R*' calculated from several injections of the test solution,

R_{std} = the mean value of the '*R*' calculated from several injections of reference standard solution, and

T = the assay expressed in percent of the standard phosalone.

A-2. SPECTROPHOTOMETRIC METHOD

A-2.1 Apparatus

A-2.1.1 *Spectrophotometer*

A-2.2 Reagents

A-2.2.1 *Absolute Alcohol*

A-2.2.2 Acetonitrite

A-2.2.3 Carbon Tetrachlorile

A-2.2.4 Standard Sodium Hydroxide — 0·5 *N.*

A-2.2.5 Standard Hydrochloric Acid — 0·1 N.

A-2.2.6 Copper Sulphate ($CuSO_4.5H_2O$) 1'5 percent (*m/v*).

A-2.2.7 *Standard Reference Phosalone*

A-2.3 Procedure

A-2.3.1 Weigh accurately 0·240 to 0·260 g of the sample into a 250-ml volumetric flask. Dissolve and make up to mark with absolute alcohol.

A-2.3.2 Take 5 ml of solution (see A-2.3.1) into another volumetric flask of 100-ml, add 25-ml of acetonitrile and make up the volume with absolute alcohol.

A-2.3.3 Take 10 ml of solution (see A-2.3.2) in a 250-ml conical flask with stopper and add 2 ml of sodium hydroxide (see A-2.2.4), agitate for 60 seconds and allow to stand for 60 minutes. Add 100-ml of hydrochloric acid (see A-2.2.5), agitate for 60 seconds and allow to stand for 5 minutes.

A-2.3.4 Add 50 ml of carbon tetrachloride and 2 ml of copper sulphate (see A-2.2.6). Agitate vigorously for sixty seconds. Separate the carbon tetrachloride phase from the aqueous phase in a dry separating funnel and filter through Whatman filter paper No. 1.

A-2.3.5 Measure the optical density at 420 my using a spectrophotometer with a 4-cm cell, the instrument being previously calibrated using carbon tetrachloride as a reference.

NOTE — After addition of copper sulphate solution, the optical density measurement should be taken in exactly five minutes.

A-2.3.6 Carry out the same tests (see A-2.3.1 to **A-2.3.5)** on standard reference phosalone (see A-2.2.7).

A-2.4 **Calculation**

A-2.4.1 Phosalone content, percent by mass $= \dfrac{M_1 \times N \times T}{S M_2}$

where

M_1 = mass of standard reference phosalone,
N = optical density of the sample,
T = percent purity of the phosalone,
S = optical density of the standard phosalone, and
M_2 = mass of the sample taken for the test.

APPENDIX B

[*Table 1, Item* **(iii)**]

DETERMINATION OF MOISTURE

B-I. VACUUM-OVEN METHOD

B-1.1 Apparatus

B-1.1.1 *Flat-Bottom Dish* — of nickel or other suitable material. 70 to 80 mm in diameter and not more than 25 mm deep.

B-1.1.2 *Vacuum Oven*

B-1.2 Procedure

B-1.2.1 Heat the dish in an oven at 105°C for about an hour. Allow to cool in an efficient desiccator for 30 to 40 minutes. Weigh accurately about 2 g of the material into a tared dish dried as above.

B-1.2.2 Transfer the dish to the vacuum oven maintained at a pressure not exceeding 5 mm of Hg and at room temperature.

B-1.2.3 After 24 hours, remove the dish to a desiccator, cool and weigh.

B-I.3 **Calculation**

B-1.3.1 Moisture content, percent by mass $= \dfrac{100\,(M_1 - M_2)}{M_1 - M}$

where

M_1 = mass, in g, of the contents of the dish before drying;
M_2 = mass, in g, of the contents of the dish after drying; and
M = mass, in g, of the empty dish.

(Continued from page 2)

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(*Continued on page* 11)

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INDIAN STANDARDS
ON
PESTICIDES (TECHNICAL GRADE)

IS:	
560-1969	BHC, technical and refined (*second revision*)
563-1973	DDT, technical (*second* revision)
1051-1973	Pyrethrum extracts (*first revision*)
1251-1973	*Zinc* phosphide, technical (*first revision*)
1306-1974	Aldrin, technical (*first revision*)
1309-1974	Endrin, technical (first *revision*)
1486-1969	Copper oxychloride, technical (first *revision*)
1682-1973	Cuprous oxide, technical (fungicidal grade) (*first revision*)
1832-1961	Malathion, technical
1833-1961	Diazinon, technical
2125-1962	Phenyl mercury salicylate, technical
2126-1973	Phenyl mercury acetate, technical (*first revision*)
2127-1962	Stabilized methoxy ethyl mercury chloride concentrate
2128-1973	Parathion ethyl, technical (first *revision*)
2353-1963	Phenyl mercury chloride, technical
2354-1963	Ethyl mercury chloride, technical
2355-1963	Stabilized ethoxy ethyl mercury chloride concentrate
2570-1963	Methyl parathion., technical
2863-1964	Chlordane, technical
3898-1966	Zineb, technical
3900-1965	Ziram, technical (*first revision*)
3904.1966	Thiometon concentrates
4320-1967	Thiram, technical
4321-1967	2, 4-D technical
4344-1967	Endosulfan, technical
4345-1967	Binapacryl, technical
4451-1967	Toxaphene, technical
4929-1968	Dichlorvos, technical
4958-1968	Phosphamidon, technical
5278-1969	Dicofol, technical
5280-1969	Fenitrothion, technical
5526-1969	Coumafuryl, technical
5551-1970	Warfarin-sodium, technical
5552-1970	Warfarin, technical
6432-1972	Heptachlor, technical
7158-1973	Isobornyl thiocyanoacetate (thanite), technical
7539-1975	Carbaryl, technical
7945-1976	Trichlorfon, technical
7950-1976	Fenthion, technical
7976-1976	Phorate, technical
7977-1976	Disulfoton, technical
7985-1976	Quintozene, technical
8024-1976	Formothion technical solution
8025-1976	Monocrotophos, technical
8071-1976	Propanil, technical
8072-1976	Quinalphos, technical
8258-1977	Oxydemetan-methyl, technical
8X7-1977	Delepan sodium, technical
8293-1977	Phenthoate, technical
8445-1977	Carbendazin (MBC), technical
8496-1977	Propoxur, technical
8488-1977	Phosolane, technical

AMENDMENT NO. 1 OCTOBER 1979

TO

IS:8488-1977 SPECIFICATION FOR PHOSALONE, TECHNICAL

Alteration

(*Page 8, clause* A-2.2.1) - Substitute the following for the existing clause:

'A-2.2.1 *Absolute Alcohol* - alternatively methyl alcohol, anhydrous may be used.'

(AFCDC 6)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 2 JUNE 1981
TO
IS : 8488 - 1977 SPECIFICATION FOR PHOSALONE, TECHNICAL

Alterations

(*Page* 4, *clause* 3.1) — Substitute the following for the existing clause:

'**3.1 Packing** — The material shall be packed according to the requirements given in IS : 8190 (Part I)-1980*.'

(*Page* 4, *foot-notes with* ' * ', ' † ', ' ‡ ' *and* ' § ' *marks*) — Substitute the following for the existing foot-notes:

'*Requirements for packing of pesticides : Part I Solid pesticides (*first revision*).'

(*Page* 5, *clause* 3.2.1) — Delete and renumber the subsequent clause ' 3.2.2 ' as ' 3.2.1 '.

(*Page* 5, *foot-note with* ' * ' *mark*) — Delete.

(*Page* 8, *clause* A-2.3.1) — Substitute the following for the existing clause:

'**A-2.3.1** Weigh the sample accurately by applying the formula:

$$\frac{A \times B}{C}$$

where

A = mass of reference standard taken (0·240 to 0·260 g),

B = percentage purity of the reference standard, and

C = declared percentage of technical (mass to be taken up to third decimal place).

Transfer the sample quantitatively to the 250 ml volumetric flask. Dissolve and make up to mark with absolute alcohol.

(AFCDC 6)

AMENDMENT NO. 3 NOVEMBER 1988

TO

IS : 8488 - 1977 SPECIFICATION FOR PHOSALONE, TECHNICAL

[*Page* 8, *clause* A-2.3 (*see also* Amendment No. 2)] — Substitute the following for the existing clause:

'A-2.3 **Procedure**

A-2.3.1 Weigh accurately 0·260 g of the sample (equivalent to 0·090 g of phosalone) in a 250-ml volumetric flask. Dissolve and make up to mark with absolute alcohol or methyl alcohol.

A-2.3.2 Take 5 ml of solution (*see* A-2.3.1) into a 250-ml separating funnel. Add 10 ml of absolute alcohol, 2·5 ml of acetonitrile and 2 ml of standard sodium hydroxide. Agitate for 60 seconds and allow to stand for 60 minutes. Add 100 ml of standard hydrochloric acid, agitate for 60 seconds and allow to stand for 5 minutes.

A-2.3.3 Add 50 ml of carbon tetrachloride and 2 ml of copper sulphate. Agitate vigorously for 60 seconds. Allow the layers to separate. Transfer the coloured carbon tetrachloride phase into an 1-cm cell after filtering through anhydrous sodium sulphate.

A-2.3.4 Measure the optical density at 420 mμ using a spectrophotometer, the instrument being previously calibrated using carbon tetrachloride as a reference.

NOTE — After addition of copper sulphate solution, the optical density measurement should be taken in exactly five minutes.

Carry out the same tests (*see* A-2.3.1 to A-2.3.4) on standard reference phosalone.

A-2.3.5 *Calculation*

(AFCDC 6)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 4 NOVEMBER 1991
TO
IS 8488 : 1977 SPECIFICATION FOR PHOSALONE, TECHNICAL

[*Page 4, Table* 1, *col* 5] — **Substitute** 'IS 6940 : 1982' for 'IS 6940 : 1973'.

[*Page 4, Table* 1, *against Sl No.* (iii), *under col 4*] — *Delete.*

[*Page 4, Table* 1, *against Sl No.* (iii), *under col* 5] — **Insert '4.1'.**

(Page 4, Table 1, *foot-note*) — **Insert** at **the end** '(*first revision*)'.

(FADC 1)

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